# AN INVESTIGATION OF THE BEHAVIOR OF THE SHEAR MODULUS OF ALUMINUM WITH ANNEALING AFTER COLD WORK AT -190°C

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## ABSTRACT

By observing the dependency of the shear modulus of aluminum upon annealing after plastic deformation at -190°C, an investigation of dislocation effects was conducted. Aluminum was selected since its shear modulus is nearly isotropic, and it was desired to correlate the results of this investigation with the results of a similar investigation of the shear modulus of copper in which the effect of anisotropy was unknown. The shear modulus of aluminum was determined by observing the period of a pendulum utilizing aluminum as the torsional element.

Cold work by twisting (surface shear strain of 0.07) lowered the shear modulus by approximately 10% of the pre-cold work value and annealing to 250°C produced an 8% recovery. Marked changes from a nearly linear relationship during recovery occurred in the regions between 0°C to 50°C and 110°C to 175°C. This behavior fits a model of a crystal lattice in which pinning points with activation energies lying near two definite values migrate out of the lattice and permit dislocation migration at definite temperatures.

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# CHAPTER I

## INTRODUCTION

Perhaps an outstanding characteristic of the field of mechanical properties of metals is the lack until relatively few years ago of concentrated effort towards a complete understanding of basic phenomena. Metallurgy has developed for centuries more as an art than a science; and man has known the basic properties of metals and how certain treatments affect these properties for thousands of years. While much progress has been made recently in determining the mechanisms which influence mechanical properties of solids, the understanding of the manner in which these mechanisms act is continually being developed.

Since 1934 when G. I. Taylor and E. Orowan introduced the dislocation theory, much progress has been made in explaining the basic mechanical properties of metals in terms of dislocations.

With the ever increasing use of nuclear energy, both militarily and industrially, an understanding of the effects of powerful sources of radiation upon metals is imperative. In order to predict the behavior of irradiated metals, a basic understanding of the mechanisms which determine their unradiated properties must be achieved.

During the past several years studies have been in progress at the United States Naval Postgraduate School with the overall objective of better understanding the basic mechanisms involved in irradiation of crystal lattices, their recovery, and the changes produced in the physical and mechanical properties of the material under study.

The investigation reported here was conducted by the authors during the 1957-58 academic year at the Postgraduate School, under the supervision of Dr. E. C. Crittenden, Jr. It had as its aim the study of dislocation effects by observing the dependency of the shear modulus of aluminum upon annealing after plastic deformation at very low temperatures. It was also hoped that these results would clarify the results of a similar study of the shear modulus of copper carried out by Dr. Crittenden and others. In short, we envisioned using dislocations as a tool to probe other phenomena, namely, the effect of point defects such as interstitial atoms and vacancies.

# CHAPTER II

## SYNPOSIS OF EXPERIMENT

### 1. Theory and Previous Work

Since the aim of this investigation is to study dislocation effects by observing the shear modulus, it seems pertinent to review some of the basic concepts of the mechanisms which influence dislocation effects.

A change in the elastic modulus of a metal can occur due to lattice distortion by point defects such as vacancies and interstitials. A vacancy is a vacant lattice site and an interstitial is an atom whose equilibrium position is at a position other than a normal lattice site. Dienes[1] has calculated that a density of one percent interstitial atoms would increase the shear modulus in the (111) plane for copper by about five percent, while a one percent density of vacancies would reduce it by one percent. The change in shear modulus in this plane is chosen here because the (111) plane proved to be the most desirable plane in which to measure the shear modulus of aluminum for reasons to be stated later. It is reasonable to assume similar moduli changes would occur in aluminum as predicted in copper.

Not only is the shear modulus affected by point defects but, to an even greater extent, by the action of dislocations whose effect masks the effect of point defects. A dislocation is a very specific type of imperfection in a crystal lattice and much has been written concerning its modus operandi. [1] [2] A dislocation is a configuration that can move through the lattice, but the motion of a dislocation is a convenient way to refer to the motions of large numbers of atoms. Each atom moves

[1]G. J. Dienes, J. Apl. Phys. 24, 666 (1953)

only one interatomic distance or less as the configuration moves through many atomic spacings.

The presence of a dislocation will lower the elastic constants of an otherwise perfect crystal. That this is true can be easily understood. The shear modulus, G, is the ratio of shear stress to shear strain. When a shear stress is applied to an ideal crystal with no dislocations present, the crystal deforms after reaching its elastic limit by planes of atoms sliding over one another like cards in a deck. With a dislocation present in the same crystal, the motion of a dislocation through the crystal produces a macroscopic deformation in which the crystal yields to applied stresses. The stress required to move a dislocation is considerably smaller than the stress required to shear one entire plane of atoms simultaneously over another in a perfect crystal.

When a very small stress is applied to a crystal containing dislocations a different phenomena occurs, and it may be thought of as a pseudo-slip. Under small stresses a dislocation will bow between fixed pinning points by motion at right angles to its length without breaking away from these pinning points. This pseudo-slip is reversible since the dislocation may reverse its direction of motion when stressed in an opposite direction. Since the presence of a dislocation in a crystal lattice introduces an additional shear strain for the same amount of shear stress, it is seen that the ratio of stress to strain (the shear modulus) will be reduced.

This shear modulus reduction effect of a dislocation is modified, however, by the pinning action of point defects such as vacancies, interstitials and impurities and the interaction of other dislocations

which all decrease the effective length of the dislocation. This effective length is the prime quantity which determines the behavior of a dislocation in an otherwise perfect crystal lattice, since it governs the amount of pseudo-slip produced by bowing of the dislocation.

The number of dislocations can be specified by n, the average number intersecting per unit area, and l, the average effective length of all dislocations. The presence of dislocations will, in general, lower the measured shear modulus. According to Mott,[2] the modulus will vary with n and l as follows:

$$G = G_0\left(1 - \frac{nl^2}{6}\right)$$

Where $G_0$ is the shear modulus of a perfect crystal without any dislocations. The presence of impurities can change the effective length of the dislocation and interfere with its motion. These impurity atoms could migrate to or be swept up by a dislocation and, in so doing, would form pinning points for the dislocation, causing it to act as a number of shorter dislocations. In effect, the l in the above equation would be reduced, causing a rise in G. In a like manner, interstitials and vacancies should migrate to or be picked up by dislocations and form pinning points with a similar effect on the shear modulus.

The production of interstitial-vacancy pairs may be achieved by bombarding a crystal with energetic electrons. Using this means of creating interstitial-vacancy pairs, an investigation of the change in shear modulus with increasing interstitials and vacancies of

[2] N. F. Mott, A Theory of Work Hardening of Metal Crystals, Philosophical Magazine, 43, 1952, page 1151

intentionally cold worked copper specimens was performed in June 1956 at the U. S. Naval Postgraduate School.[3] In these experiments the modulus decreased upon cold working and increased during irradiation with 2 Mev electrons. In all cases G, as a function of integrated electron flux, increased during irradiation in roughly an exponential manner up to a constant value, reached after a 1% increase in the case of absolute minimum cold work, a 10% increase for 190° twist, and a 14% increase for one full turn. Following irradiation, specimens were annealed from -195° C up to +300° C for 15 minutes at each increment of 25° C. In all cases G remained approximately constant during annealing. Hitherto unpublished plots of shear modulus vs. integrated electron flux obtained during this investigation are presented in Figures 8 and 9. This behavior of increased G with increasing electron flux fits a model employing stable pinning of dislocations by interstitials and vacancies. In terms of Mott's theory, n remains constant and l decreases with the introduction of pinning points; hence a rise in modulus will follow.

It is well known that cold working will bring about a change in the shear modulus, but the manner in which this change takes place is not fully understood. There is now much evidence which indicates that cold working not only multiplies dislocations but introduces interstitials and vacancies as well. The multiplication of dislocations with plastic deformation is now accounted for in terms of the Frank Read mechanism.[4] Plastic deformation of a lattice ensues where the value of shear stress is such to produce slip in the slip plane. Dislocations then multiply

[3] D. A. Powell, H. S. Sellers, E. A. Milne and E. C. Crittenden, The Effects of Electron Irradiation on the Shear Modulus and Internal Friction of Copper, Bulletin of the American Physical Society, Vol 1, No. 8, 27 Dec 1956, page 379

[4] W. T. Read, Jr., Dislocations in Crystals, McGraw Hill Book Company, New York (1953), p 69

by the Frank Read mechanism. With excessive slip, the dislocations interact by crossing and commotion results when two dislocations pull thru one another. Vacancies and some interstitials may be formed in this process. Vacancies are easier to create than interstitials since more energy is required to form an interstitial. When two intersecting dislocation lines cut through each other, the result depends upon their orientations and Burgers vectors. There are several possibilities, but the only one which has received much attention so far is the crossing of two screw dislocations, because this leads to the generation of vacant sites or interstitial atoms.[5] According to another source, an edge dislocation is both a source and a sink for vacancies and interstitial atoms.[6] It is clear however that dislocations can generate and annihilate these two types of point defects.

In 1953, Crittenden and Dieckamp[7] studied in some detail the behavior of the shear modulus of (111) oriented copper after cold work by twisting at various annealing temperatures. All the values of shear modulus were measured at -196° C. The results of this work, though somewhat inconclusive, did show a marked recovery in the shear modulus following annealing. An interesting phenomena also shown was the degree of recovery of shear modulus wherein marked rises and plateaus were formed at definite temperature intervals. Figures 10 and 11 show the results of their work with specimens of surface shear strains comparable to the value of shear strain employed in this investigation with aluminum. Zones of marked rise in G were even more pronounced with higher values of

[5]A. H. Cottrell, Dislocations and Plastic Flow in Crystals, Clarendon Press, Oxford, 1953, p. 172

[6]Ibid, Read, p. 49

[7]E. C. Crittenden, Jr., and H. Dieckamp, Phys. Rev 94, 1418 (1954)

shear strain. The same rises and plateaus were found in the G of specimens cold worked by elongation.

Crittenden and Dieckamp have since explained these rises in terms of the migration of pinning points and dislocations out of the crystal during annealing. They believed that the cause of the rise was due to a reduction of n, the average number of dislocations per unit area. Motion of particular types of pinning points will be possible in certain temperature zones depending upon their activation energy. Their motion will permit dislocations to leave the lattice and produce rapid rise regions in the recovery curve. Dislocation movement is relatively small in the plateau regions because the remaining pinning points are fairly permanent until a temperature high enough to activate their migration is reached. The first initial rise in G was attributed to a straightening out and unsnarling of dislocations and decrease in n as dislocations migrated out of the crystal rather than to migration of a certain type of pinning point.

The formation of the steeper portions of the recovery curve, however, could also be due to the anisotropic nature of copper. Recrystallization and rearrangement could account for the increase in G since a (111) orientation produces the lowest possible modulus and any rearrangement could only increase the value of G.

During annealing one of the frequently proposed mechanisms of recovery is the migration of interstitials and vacancies with their disappearance and or annihilation of close pairs. The migration of interstitials occurs at a lower temperature than vacancy migration. There are two possibilities to account for the disappearance of these point defects. They could migrate out of the crystal lattice and to crystal boundaries or there is the possibility that they may migrate to

dislocations. If they should migrate to a dislocation, this would cause portions of the dislocation to climb, forming notches. These notches might have a low resistance to motion along the dislocation; however, the undisturbed portions of the dislocation would have an increased resistance to motion in the slip plane. In effect, glide in the slip plane would be restricted by the pinning action of the vacancies and interstitials, hence causing an increase in the shear modulus.

There is another possibility to account for the recovery of the shear modulus upon annealing. This would involve the migration and disappearance of dislocations from the crystal lattice.

## 2. Concept of Experiment

The rises exhibited by copper in the study of Crittenden and Dieckamp were explained by them using a model of migrating dislocations and pinning points; however, the rises in the curve may have been due to crystal reorientation. If a similar test, conducted with an isotropic specimen, produced these same rises and plateaus, then anisotropy could be ruled out as a factor in their formation and support for the model would be obtained.

Aluminum, being essentially isotropic, was selected to eliminate the possibility of anisotropy effect. Extremely high purity aluminum was considered essential to eliminate the effects of impurities acting as pinning points for dislocations. For isotropic behavior it would have been optimum to have an aluminum single crystal specimen with a [111] orientation parallel to the axis of the wire, since crystal boundaries may impede dislocation movement. The first problem, then, was to obtain

the desired aluminum specimens. The methods used and the results obtained will be covered in Chapter III.

In view of the difference between the melting points of copper (1083° C) and aluminum (659.7° C) and the fact that the copper investigation was made at liquid air temperatures, it was expected that the same relative modulus variations in aluminum would be observed in a temperature region lower than that of liquid air. With this in mind, the equipment for observing the shear modulus was designed and built for use in the liquid helium range. The basic apparatus, a torsion pendulum assembly, was already present, having been utilized in previous experiments in this field. The problem, as far as equipment was concerned, consisted of modifying and adapting available equipment for use at very low temperatures. How this was done and a description of the equipment will be presented in Chapter IV.

In the performance of this experiment, it was desired to introduce cold work in and observe the shear modulus of the specimen following successive anneals at liquid helium temperature. During preliminary testing, the liquid helium dewar flask developed a crack and was useless for further work. The delay in procuring a new flask dictated that data be obtained in the liquid air range. Consequently, this investigation was conducted at liquid air temperature and results are reported in Chapter VI.

## CHAPTER III

## SPECIMEN

### 1. Choice of Specimen

The question of the possibility of complications due to anisotropy in the case of copper needed to be settled. Aluminum was chosen as the means of resolving this uncertainty since its shear modulus is nearly isotropic regardless of the direction of slip. All cubic crystals such as copper and aluminum are exactly isotropic for all directions in the (111) plane; and since the behavior of copper in this plane was investigated, it was desired to do the same with aluminum. The lowest possible value of the modulus also occurs in this plane; hence, any change in the modulus after cold working of a (111) oriented single crystal should be entirely dislocation induced. It was, however, recognized that crystal growth with high purity aluminum would be difficult to achieve.[1,2] The anisotropy factor of a crystal is defined[3] as

$$A = \frac{2C_{44}}{C_{11} - C_{12}}$$

where $C_{11}$, $C_{12}$ and $C_{44}$ are the customary elastic constants. A perfectly isotropic crystal would have an A value of one whereas copper and aluminum have values of 3.3 and 1.23 respectively. If reorientation by recrystallization had occurred with copper during pulse annealing below room temperature, then the results of aluminum should differ from those of copper.

[1] A. V. Seybolt and J. E. Burke, Procedures in Experimental Metallurgy, John Wiley and Sons, New York, 1953, p. 323

[2] E. Schmid and I. W. Boas, Plasticity of Crystals, Hughes and Company, English Translation, 1950, p. 25

[3] C. Zener, Elasticity and Anelasticity of Metals, University of Chicago Press, 1948, p. 41

The requirements of the specimen were considered to be the following:

a. A fiber orientation with the [111] direction parallel to the axis of the specimen for isotropic behavior.

b. A single crystal to eliminate the uncertainty of the effect of crystal boundaries upon the movement of dislocations.

c. A crystal approximately two inches long to fit into existing apparatus.

d. A constant cross section throughout the specimen length for meaningful evaluation of the shear modulus.

2. Background

When a polycrystalline metal is deformed plastically, the orientation of individual grains is altered toward a preferred orientation, wherein certain lattice directions are aligned with the principal directions of flow in the metal. This alteration of orientation is not complete until the metal has received a cross-section reduction of 90% or more according to Barrett;[4] and, in the case of aluminum, this fiber texture after cold working is 100% of crystals with [111] parallel to the wire axis.

After deformation and removal of external stresses, some of the internal stresses produced during deformation remain. These stressed regions possess a higher energy than the unstressed regions and are thermodynamically unstable relative to the unstressed regions. A transition from the stressed to unstressed state is permissible thermodynamically and should take place in the course of time if the temperature is sufficiently high. This process, known as recrystallization, then, consists in the nucleation and growth of strain-free grains out of

[4] C. S. Barrett, Structure of Metals, McGraw Hill Book Co., New York, 1952, pp 442 and 443

the matrix of cold worked metal. Textures in aluminum wires after recrystallization have been found[5] as follows:

"1. No alteration of the deformation texture, which is [111] , with recrystallization below $500^\circ$ C.

"2. Increasing randomness with recrystallization above about $500^\circ$ C, particularly with wires of lower purity.

"3. A new texture [112] in 99.95% aluminum recrystallized at $600^\circ$ C.

"4. A sharpening of the deformation texture in 99.95% wire recrystallized at $600^\circ$ C.

"5. Retention and sharpening of the [111] component (in wire that had been drawn 99.7%) at $500^\circ$ C and conversion to [210] after coarsening at $630^\circ$ C."

The recrystallization temperature for aluminum (99.999%) has been listed as $175^\circ$ F ($79.4^\circ$ C) and is defined as the temperature at which the highly cold-worked metal completely re-crystallizes in about one hour.[6]

After an extreme reduction in cross section, the shear modulus is lowered markedly, and it is desirable to anneal the specimen to increase the modulus to what it was prior to deformation. At the same time, it is necessary to preserve the [111] texture for isotropic behavior. The recrystallization texture on completion of anneal will be essentially that stated above and should closely resemble the deformation texture from which it grew.

[5] Ibid, p. 486

[6] A. G. Guy, Elements of Physical Metallurgy, Addison-Wesley Press, Inc., Cambridge, Mass., 1951, p. 226

3. Production of Specimen

Crystal growth from the melt was not considered feasible in view of fiber-orientation and constant cross-section requirements. Johnson Mathey aluminum wire of 99.999+% purity and 0.25 inch diameter was deformed by cold drawing to a diameter of 0.0442 cm. by 70 draws through a plate wire die. The final diameter was equivalent to a 99.7% reduction in cross sectional area.

It was decided to attempt crystal growth utilizing the "strain-anneal" method with the technique employed by Andrade,[7] since a vacuum furnace with moving gradient constructed for a project[8] at the U. S. Naval Postgraduate School was available. After an initial anneal of 300° C, strains of 1-4% were tested at varying annealing temperatures and specimen velocities through the furnace. Results were somewhat disappointing in that the maximum crystal size achieved was three times the wire diameter. These were obtained by a 650° C final anneal at the slowest specimen velocity of 0.005 mm/sec through a 10 cm. furnace at pressure of $1.5 \times 10^{-5}$ mm of mercury. No difference in crystal size could be attributed to varying strain.

Further crystal growth attempts were made using the strain-anneal method of the Aluminum Company of America.[9] Greater success was achieved

[7] A. V. Seybolt and J. E. Burke, Proceedings in Experimental Metallurgy, John Wiley and Sons, New York, 1953, p. 320

[8] H. D. Peckam and H. C. Kinne, Jr., Oriented Single Crystal Copper Fibers by a Strain-Anneal Technique, Thesis, U. S. Naval Postgraduate School, Monterey, California, 1957

[9] A. D. Schwope, F. R. Shober and L. R. Jackson, Creep in Metals, NACA Technical Note 2618, National Advisory Committee for Aeronautics, Washington, D. C., 1952

with this method. A strain of one percent produced a crystal 0.65 inches in length; however, this was not long enough for use in the torsional pendulum equipment.

In view of the failure to produce a single crystal specimen, a cold work-anneal method was used to produce a polycrystalline wire which showed a [111] fiber orientation verified by X-ray diffraction. A typical specimen was etched and observed under a microscope. The following formula was most satisfactory in producing an etch which would reveal the grain structure:

3 parts HF

47 parts $HNO_3$

50 parts HCL

The average crystal size of the wire was one to two times diameter and was produced as described above without the strain phase.

## CHAPTER IV

## EQUIPMENT

### 1. General Considerations

In the design of the equipment utilized in this project, four major factors were taken into consideration:

a. A means of externally introducing cold work into the test specimen.

b. Provisions for introducing and maintaining liquid helium within the system without disturbing the test specimen.

c. An accurate control of the annealing furnace temperature and measurement of the specimen temperature.

d. Provisions for externally initiating and counting the oscillations of the torsion pendulum.

The equipment was constructed to be operated at either liquid helium or liquid air temperatures. Operation with liquid helium involved the addition of the styrofoam liquid air jacket (Figure 2) and use of the carbon composition resistor (Figure 5) for temperature measurements in the region -268.8° C to -200° C.

As far as possible equipment utilized in previous work in this field was adapted and modified to fit the needs of this project.[1]

### 2. Basic Equipment

Figure 1 gives a schematic display of the essential elements of the torsion pendulum and associated dewar flasks. Figure 2 shows the overall arrangement of the equipment.

[1]D. A. Powell, Jr., and H. S. Sellers, The Effects of Electron Damage on the Shear Modulus of Copper, Thesis, U. S. Naval Postgraduate School, 1956

The torsion pendulum and inner dewar flask were supported from a metal plate which was attached to the wall. The front portion of this plate incorporated a plexiglass window for passage of the light beam used to measure the period of the pendulum. This metal plate and associated equipment provided a good pressure seal for the inner dewar flask. For liquid helium use it was first necessary to precool the inner dewar with liquid air, purge the system of the liquid air by pressurizing with helium gas, and then introduce the liquid helium. In order to maintain the temperature as low as possible during this purging process, it was found necessary to precool the gaseous helium through a pre-cooler (Figure 2) which consisted of a copper moisture trap and a coil of some twenty feet of copper 1/4 inch O. D. tubing contained in a dewar flask filled with liquid air. The pressure seal between the support plate and inner dewar flask also offered an added advantage of providing a good barrier against moisture within the inner dewar flask. It was essential that moisture not enter into this region since condensation by ice coating would affect the period of the pendulum and fog the optical system. It was found that adequate protection against moisture was offered for as long as 72 hours.

In the event of any spillage of either liquid helium or air into the torsion assembly area, a purging tube and a small resistance heater were placed immediately below this area (Figure 1) for local purging. The specimen and torsion pendulum were suspended from the central support rod (Figure 4). This rod could be externally controlled as to height and angular position. By rotating this rod, the desired amount of cold work was introduced into the specimen. The specimen was mounted

between the central support rod and the torsion pendulum by the use of cut down size 0 pin vises. Figure 5 shows the torsion pendulum with the specimen acting as the torsion element. A value of 0.104 $kgm/mm^2$ was taken as the critical shear stress of high purity aluminum at room temperature.[2] The pendulum with its pin vise weighed 19.3 grams. In a face centered cubic slip will occur along the faces of the possible (111) planes and in the $[10\bar{1}]$ direction.[3] With this criterion it was found that a weight of 57 grams could be tolerated by our specimens. Once cold work had been introduced into the specimen, slip no longer became a critical factor; and being well within the maximum weight limitations, it is considered unlikely that any slip occurred. Examination of specimens following runs failed to reveal any evidence of slip.

Oscillations of the pendulum were initiated by external movement of a large magnet reacting on a stub bar magnet, mounted normal to and through the shank of the pendulum.

3. <u>Temperature Control and Measurement</u>

The furnace, shown in the raised position (Figure 4) was a 25 watt, ceramic resistor. It was so constructed that it could be raised while mounting the specimen and then lowered and remain over the specimen for the entire run. A variac attached to a 600 volt transformer was used to control the voltage applied to the furnace. The upper cross piece holding the furnace in line was hollow and opened into a stainless steel tube extending up through the top plate. Upon completion

[2] F. D. Rosi and C. H. Mathewson, Trans, AIME, Vol. 188, 1950, page 1160

[3] C. S. Barrett, Structure of Metals, McGraw Hill, New York, 1952, p. 337

of a pulse anneal, this tube was uncorked and the main exhaust tube was closed (Figure 2) which aided in decreasing the time required to cool-down from the pulse anneal.

Temperature measurements of the specimen were taken by means of a carbon composition resistor in the $-268.8^{\circ}$ C to $-200^{\circ}$ C range[4] and by an iron-constantan thermocouple for measurements above $-200^{\circ}$ C.

An Allen Bradley 0.1 watt, 68 ohm carbon composition resistor was incorporated into a highly sensitive Wheatstone bridge circuit. In calibrating this resistor, it was found that there was a lack of reproducibility from run to run; however, reproducibility during a single run was satisfactory. Hence, when utilizing the carbon composition resistor, calibration was made at liquid air temperature and liquid helium temperature during the cooling down process of the equipment. This method led to satisfactory results during any one particular run. The accuracy of temperature measurements, utilizing this resistor, were of the order of $\pm 0.002^{\circ}$ at $-268.8^{\circ}$ C increasing to $\pm 3.6^{\circ}$ at $-200^{\circ}$ C.

The iron-constantan thermocouple, for temperature measurements above $-200^{\circ}$ C, had its "cold junction" maintained at $0^{\circ}$ C. The temperature -E.M.F. values used were obtained from a standard calibration table[5] corrected for local conditions. A Rubicon potentiometer was employed to measure the E.M.F.

[4] J. R. Clement and E. H. Quinnell, "The Low Temperature Characteristics of Carbon Composition Thermometers," Rev. of Scientific Instruments, Vol 23, 1952, page 213

[5] Handbook of Chemistry and Physics, 36th Ed., 1954-55, Chemical Rubber Publishing Co, page 2371

4. Counting System

The oscillations of the torsion pendulum were observed by utilizing a beam of light focused upon the concave mirror mounted on the pendulum (Figure 6). Figure 4 shows the 45 degree mirror which served the dual purpose of directing the incident beam of light onto the concave mirror and passing back the reflected beam onto a graduated scale. A reticle in the lamp provided a hairline for accurately determining the oscillations of the pendulum. A crystal-controlled timer (Hewlett-Packard Electronic Counter Model 522B) was employed for accurate measurement of the time. The period of the oscillation was determined by averaging the time for 100 periods. It was noted after cold work and for several of the initial low temperature anneals that a maximum of only 50 periods could be observed, due to the damping introduced by internal friction. The accuracy of this visual method of period determination is considered to be five parts in ten thousand, except for the points where only 50 periods could be observed, where the accuracy is considered to be one part in a thousand.

# CHAPTER V

## EXPERIMENTAL PROCEDURE

### 1. Preparation Phase

In order to minimize the possibility of introducing cold work while mounting the specimen in the torsion pendulum assembly, a preparation jig and mounting yoke were employed.

The preparation jig (Figure 7) was utilized while mounting the specimen in the pin vises. With one pin vise securely clamped in the preparation jig, a six centimeter, unetched, wire specimen was carefully inserted and centered in the jaws of the pin vise. While the second pin vise was loosely held in the preparation jig, the other end of the specimen was carefully positioned. The second pin vise was clamped in the jig and then both pin vises were closed. Care was taken not to bend, twist or distort the specimen during this operation.

Following mounting in the preparation jig, the length of the specimen was measured using a microscope comparator. The length of the specimen was taken to be that part of the wire between the outermost tips of the pin vises. There was a slight uncertainty as to the effective specimen length due to the failure of the pin vise jaws to close evenly over the specimen. This uncertainty in length, however, would only become important if the absolute shear modulus was desired.

After the length was determined, the specimen and pin vises were transferred to the mounting yoke (Figure 7), used for placement and securing the specimen in the torsion pendulum assembly.

By adjustment of the central support rod, the pendulum was positioned on the damping magnet (Figure 4); then the mounting yoke was removed.

From this point on, the pendulum was mainly supported by the damping magnet which also eliminated oscillation of the pendulum until specimen and pendulum were raised by means of the adjusting nuts (Figure 3).

The furnace was lowered over the specimen, care being taken to properly position the thermocouple and carbon composition resistor so as not to bear against the specimen.

Following the positioning of the inner dewar flask, the specimen and pendulum were raised clear of the damping magnet.

By means of the upper plate leveling screws (Figure 3), the pendulum was centered over the damping magnet. This was essential to reduce transverse motion when inducing rotation of the pendulum.

The apparatus was then cooled to the desired temperature, either liquid air or helium acting as the coolant.

The period of the torsion pendulum is related to the shear modulus, G, of the torsional element by:

$$G = \frac{8\pi I \ell}{r^4 \tau^2} \qquad \text{dynes/cm}^2$$

where $I$ is the moment of inertia if the pendulum, $\ell$ and $r$ the length and radius of the element and $\tau$ the period in seconds. In the above equation, $I$ was determined experimentally to equal 122 gm-cm$^2$, $r$ and $\ell$ of the torsional element were measured by means of a precision micrometer and microscope comparator respectively, and $\tau$ was determined by averaging the time for 100 oscillations of the pendulum. With considerable practice, it was found that the timing of 100 oscillations was reproducible to within .05 seconds, by manual control of the crystal timer.

The actual oscillatory motion of the pendulum was held to a minimum, consistent with the requirements for accuracy of measurement. A 2.5 centimeter swing of the light beam on the scale was sufficient for counting accuracy and this displacement resulted in a maximum shear stress in the average specimen of not over $3.3 \times 10^7$ dynes/cm$^2$. Using a value of 0.421 kg/mm$^2$ [1] or $4.13 \times 10^7$ dynes/cm$^2$ as the critical shear stress of high-purity aluminum at liquid nitrogen temperature, the elastic limit should not have been exceeded. This factor was of critical interest only for the first measurement prior to the introduction of cold work.

2. Cold Work Phase

Cold work was introduced into the specimen by manual rotation of the central support rod with the pendulum ends against the stops. It was noticed that rotation slightly in excess of the desired angle of twist, $\theta$, was necessary to compensate for recovery of the specimen. The specimens were twisted to obtain $\theta = 360°$, corresponding to a surface shear strain, $\frac{r\theta}{\ell}$, of approximately 0.07.

Upon introducing a small amount of cold work in a specimen, there is an initial decrease in the shear modulus; however, a point is reached when further cold work produces a recovery in the value of the shear modulus. An explanation for the rise in shear modulus with excessive cold work is the interaction of the numerous dislocations being formed in the specimen. In effect, such a great

[1] F. D. Rosi and C. H. Mathewson, Trans AIME, Vol 188, p. 1161 (1950)

number of dislocations are present that self-pinning, or pinning action between dislocations, occurs. This action decreases the effective length of the dislocations, causing a rise in the shear modulus. To obtain an approximate plot of this process, one specimen was subjected to various amounts of twist at liquid helium temperature. Figure 12 shows a plot of $\ell/\tau^2$, which is proportional to G, vs. $\theta$

From this graph it may be seen that for $\theta=360^\circ$, there was a marked lowering in the value of the shear modulus, without introducing the recovery associated with excessive cold work.

When introducing cold work by twisting there is a slight change in the value of the radius of the specimen. As the absolute value of shear modulus varies with $1/r^4$, any change in the radius greatly affects the absolute value of shear modulus. However, relative values on any one specimen during a run do not suffer from this difficulty.

3. Anneal Phase

After the introduction of cold work into the specimen, the period was measured at -190° C following each 15 minute pulse-anneal. Pulse-anneals were performed at successively increasing temperatures from -190° C to 250° C in increments of 25°. On each pulse-anneal, the specimen was heated and cooled as rapidly as possible. It did require, however, on the order of 12 minutes to return the specimen to -190° C after performing an anneal above 100° C.

Variations in the temperature during the pulse anneals are estimated to be less than ±1° below 0° C increasing to ±5° above 200° C. This variation in temperature was due to the difficulty in maintaining a steady

state condition and to a small lag between thermocouple reading and true specimen temperature. Also, above $100^{\circ}$ C it was much more difficult to reach anything approaching a steady state condition during the 15 minute anneal.

It should be emphasized that throughout the performance of a run every effort was made to eliminate the possibility of introducing any cold work, other than the amount originally put into the specimen.

On one of the initial trial runs erratic results were obtained which were attributed to the introduction of cold work by a slight jarring of the torsion pendulum system. On subsequent runs this difficulty was eliminated.

# CHAPTER VI

## EXPERIMENTAL RESULTS

### 1. Theory

The shear modulus, G, sometimes called the modulus of rigidity, is defined as the ratio of shear stress to shear strain. For a face-centered cubic, the shear modulus in any given direction is[1]

$$\frac{1}{G} = S_{44} + 4\left[(S_{11} - S_{12}) - \tfrac{1}{2} S_{44}\right] \Gamma \quad \text{where}$$

$$\Gamma = \alpha^2\beta^2 + \beta^2\gamma^2 + \gamma^2\alpha^2$$

and $\alpha$, $\beta$ and $\gamma$ are the direction cosines of the orientation vector. Zener[2] gives the following values of elastic constants for aluminum at room temperature:

$s_{11} = 1.59 \times 10^{-12}$; $s_{12} = -0.58 \times 10^{-12}$; $s_{44} = 3.52 \times 10^{-12}$ cm$^2$/dyne.

The value of the orientation function $\Gamma$ in the (111) plane has a constant value of 0.250. From these data a theoretical value of G is computed to be $2.54 \times 10^{11}$ dynes/cm$^2$.

It has been shown and confirmed[3] that the shear modulus of non-ferrous metals are in agreement with the following relationship:

$$G/G_o = 1 - (T/T_m)^2$$

where $G_o$ is the modulus at absolute zero and $T_m$ the melting point. From the room temperature value, it can be shown that $G_o$ will be $2.82 \times 10^{11}$ dynes/cm$^2$ and that the expected value at -190° C would be $2.77 \times 10^{11}$ dynes/cm$^2$.

[1] E. Schmid and I. W. Boaz, Plasticity of Crystals, F. A. Hughes & Co, Ltd., London, 1950, p.21

[2] C. Zener, Elasticity and Anelasticity of Metals, University of Chicago Press, 1948, p. 17

[3] Ibid, p. 155

2. Results

The values of absolute shear modulus obtained during the course of this investigation had an average value of $2.65 \times 10^{11}$ dynes/cm$^2$ and the relative values of $\ell/\tau^2$ are considered valid. The absolute shear modulus in dynes/cm$^2$ can be computed by multiplying the value of $\ell/\tau^2$ in cm/sec$^2$ by the constant $\frac{8\pi I}{r^4}$ which equals $1.2854 \times 10^{10}$ gm/cm$^2$.

The results of the cold working at -190° C and recovery with annealing for each of two specimens are shown in Figures 13 and 14. Each point on these plots is the average of from three to six successive measurements by the procedure outlined in Chapter V. Deviation of period measurements was normally about 0.05 seconds or less, and it is felt that all points are equally valid.

Figure 12 shows the variation at liquid helium temperature of shear modulus for varying degrees of shear strain. It is interesting to note that somewhere between one and two turns the reduction in shear modulus ceased and further deformation led to an increase. These data provided the basis for selecting one turn (shear strain of approximately 0.07) for subsequent runs. The modulus increase which takes place between one and two turns may be due to the locking of dislocations by other dislocations.

Figure 15 is plotted in terms of $G/G_0$ vs temperature where G is the observed value of shear modulus at each temperature. Both specimens showed about a 10% drop in modulus immediately after cold working and returned to about 98% pre-cold work value and remained steady after 175° C. A linear relationship seems to exist between -190° C and 0° C

and from $50^\circ$ C to $110^\circ$ C. Just what caused the marked change from a well established relationship in the regions between $0^\circ$ C to $50^\circ$ C and $110^\circ$ to $175^\circ$ C is not clear at this time. It is interesting to note that the slopes of the linear regions are approximately the same. Specimen one gave a larger modulus decrease after cold working due to its larger value of shear strain.

Results can be summarized as follows:

1. Cold working by twisting (shear strain of 0.07) lowered the shear modulus to approximately 90% of pre-cold work value.
2. Annealing to $250^\circ$ C produced a recovery to 98% of pre-cold work value.
3. There is not a large immediate recovery with annealing in the shear modulus of cold worked aluminum as there is with copper of comparable shear strain and in the same annealing temperature range.
4. No plateaus were found in the aluminum recovery curve as compared with the recovery curves of copper.

## CHAPTER VII

## CONCLUSIONS

Several conclusions may be drawn. Either the rises exhibited in the recovery of copper are due to its anisotropic nature, or the temperature range of observation of the recovery of aluminum was not low enough to definitely attribute the rises in copper to anisotropy, i. e., the rises may possibly be found in aluminum at lower temperatures.

For best comparison with copper results, any future observations with aluminum should be conducted in a temperature range from -190° C to -268.8° C and at varying shear strains.

Obviously there still remain many explanations to account for the behavior pattern exhibited by aluminum. Any attempt to separate the processes occurring—interstitial-vacancy anihilation, interstitial-vacacny migration to dislocations or dislocation migration—will be attended with difficulty.

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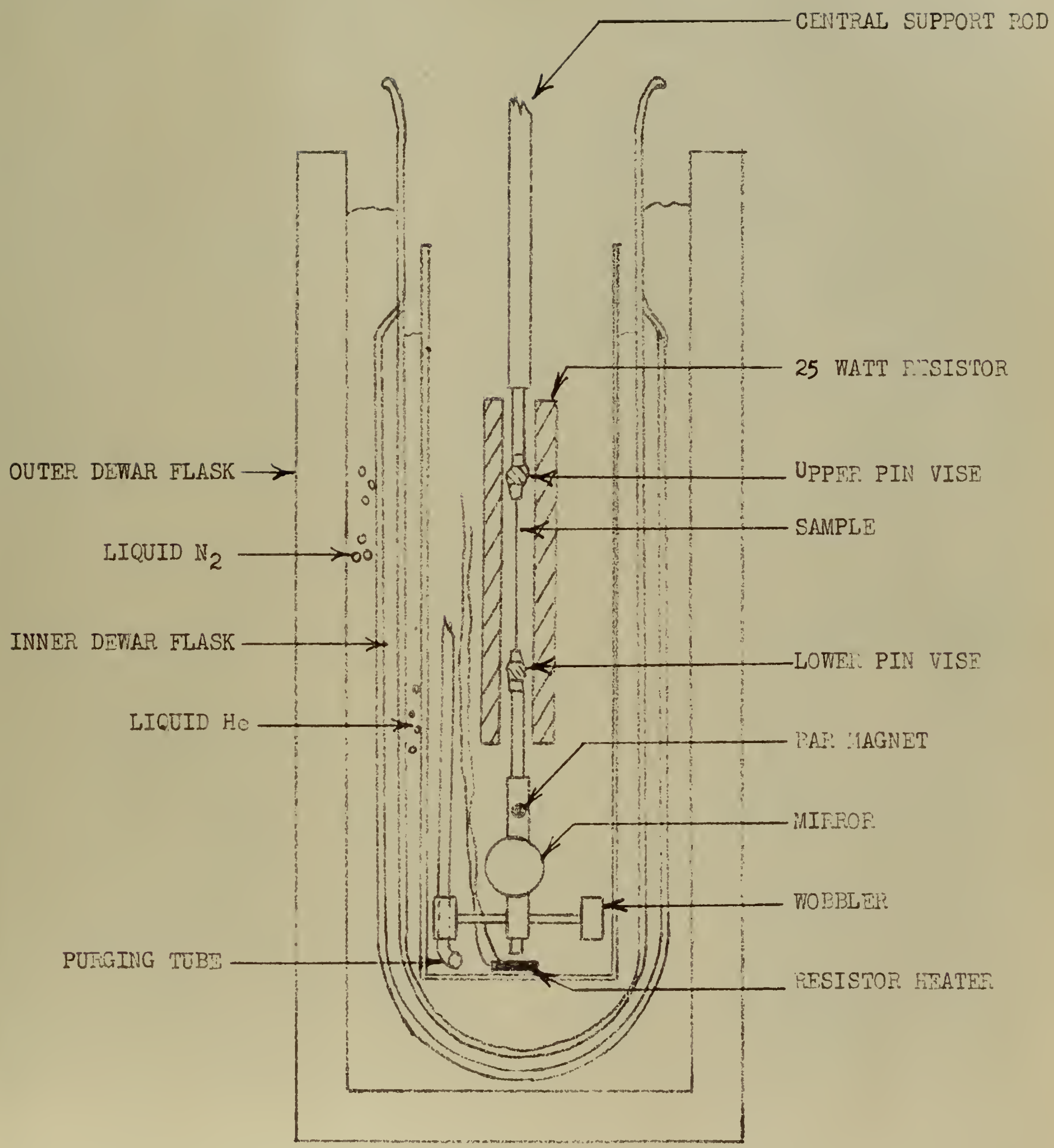



TORSION PENDULUM ASSEMBLY

FIG. 1

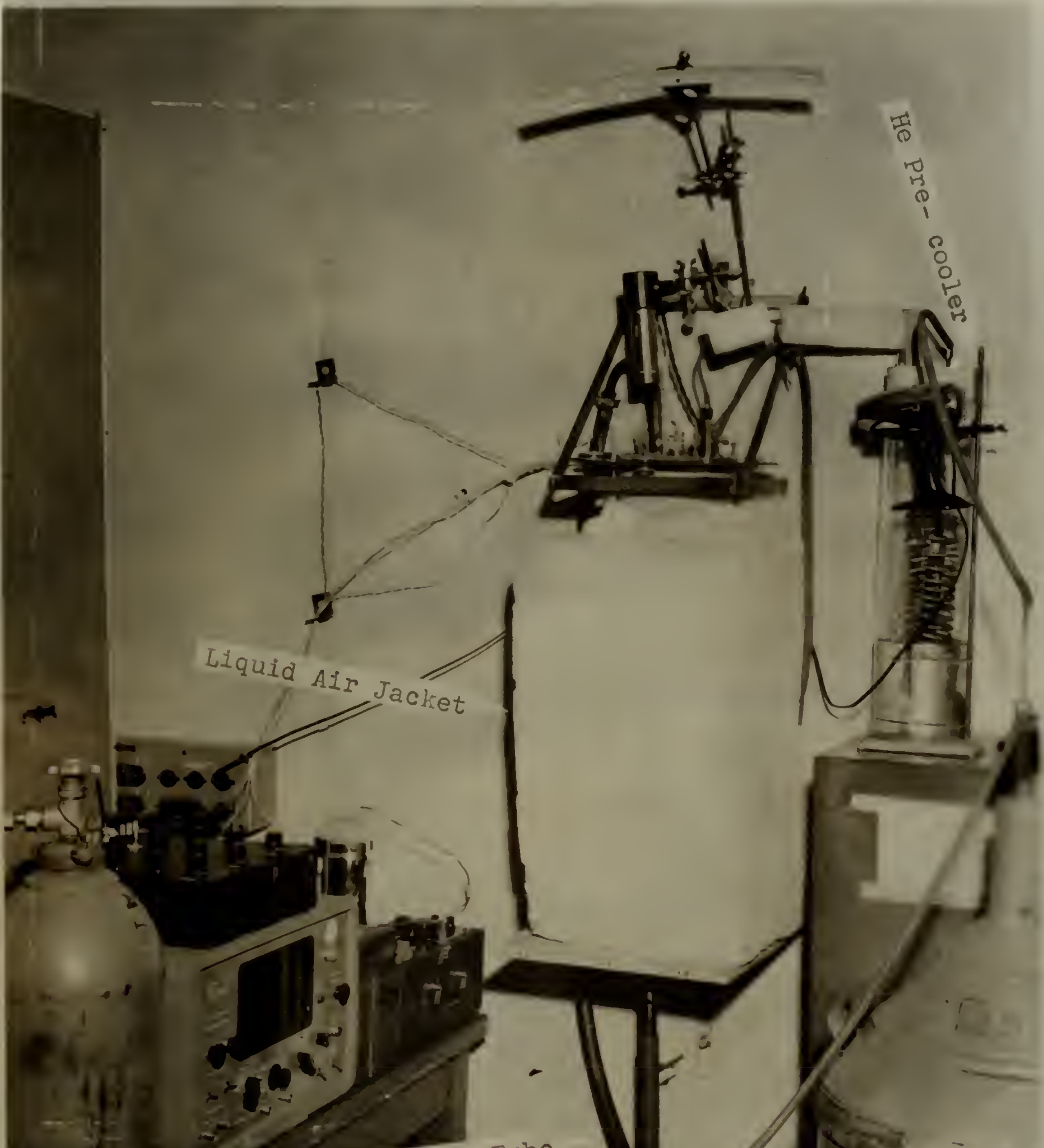



FIG. 2

GENERAL ARRANGEMENT OF EQUIPMENT


FIG. 3

TOP VIEW OF EQUIPMENT


FIG. 4

PENDULUM SUPPORT ASSEMBLY

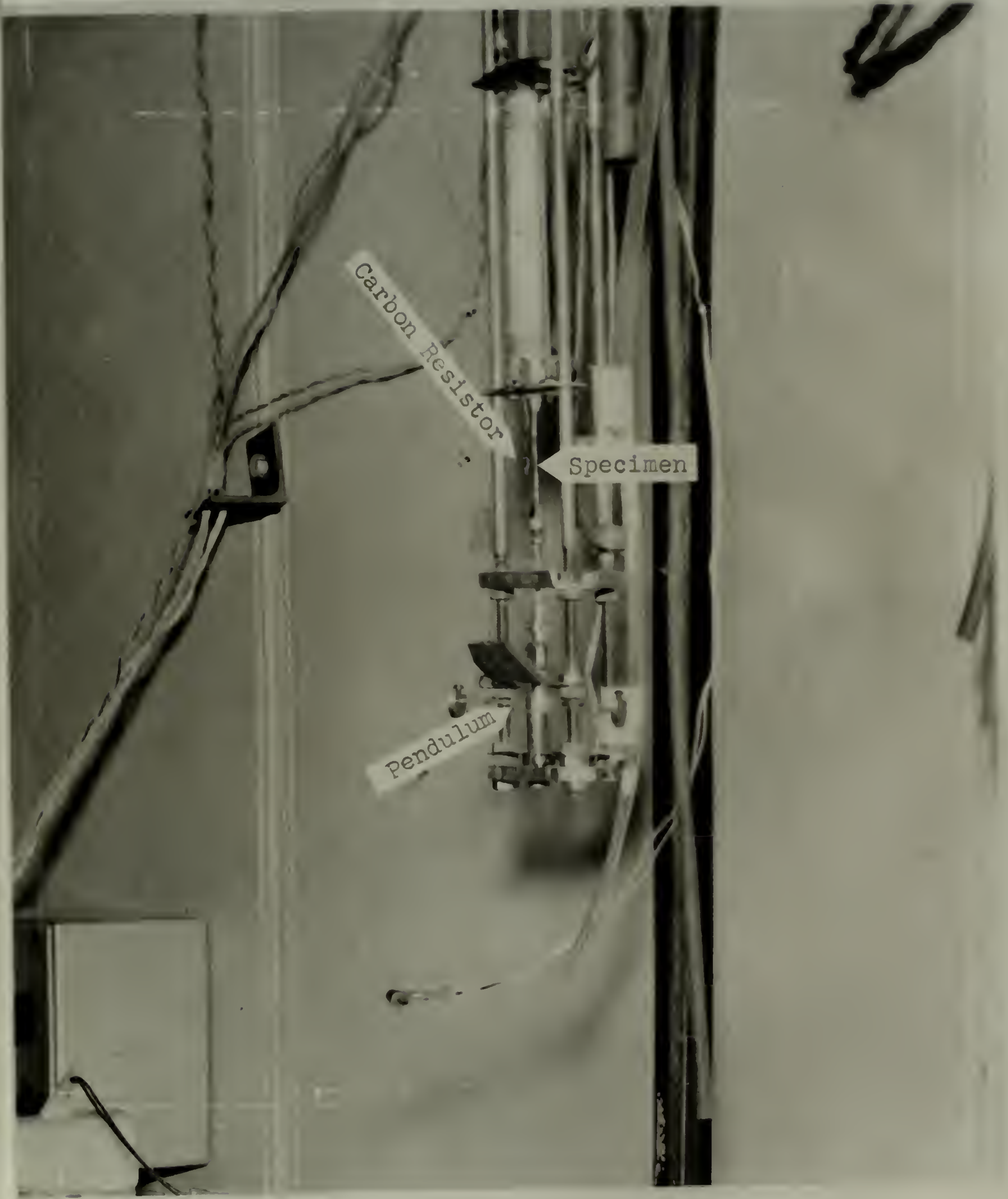



FIG. 5

CLOSE-UP of TORSION ELEMENT

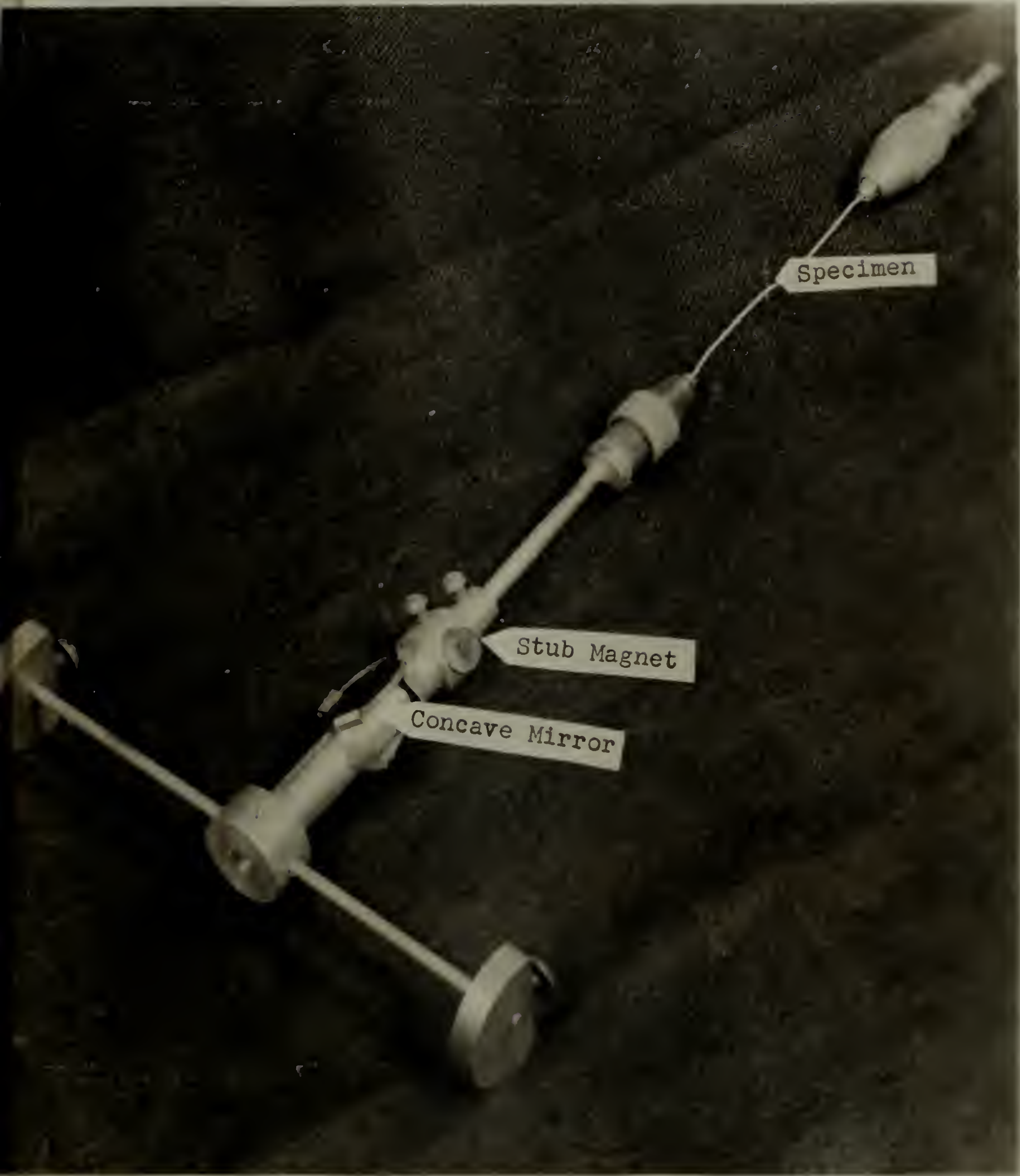



FIG. 6

TORSION PENDULUM

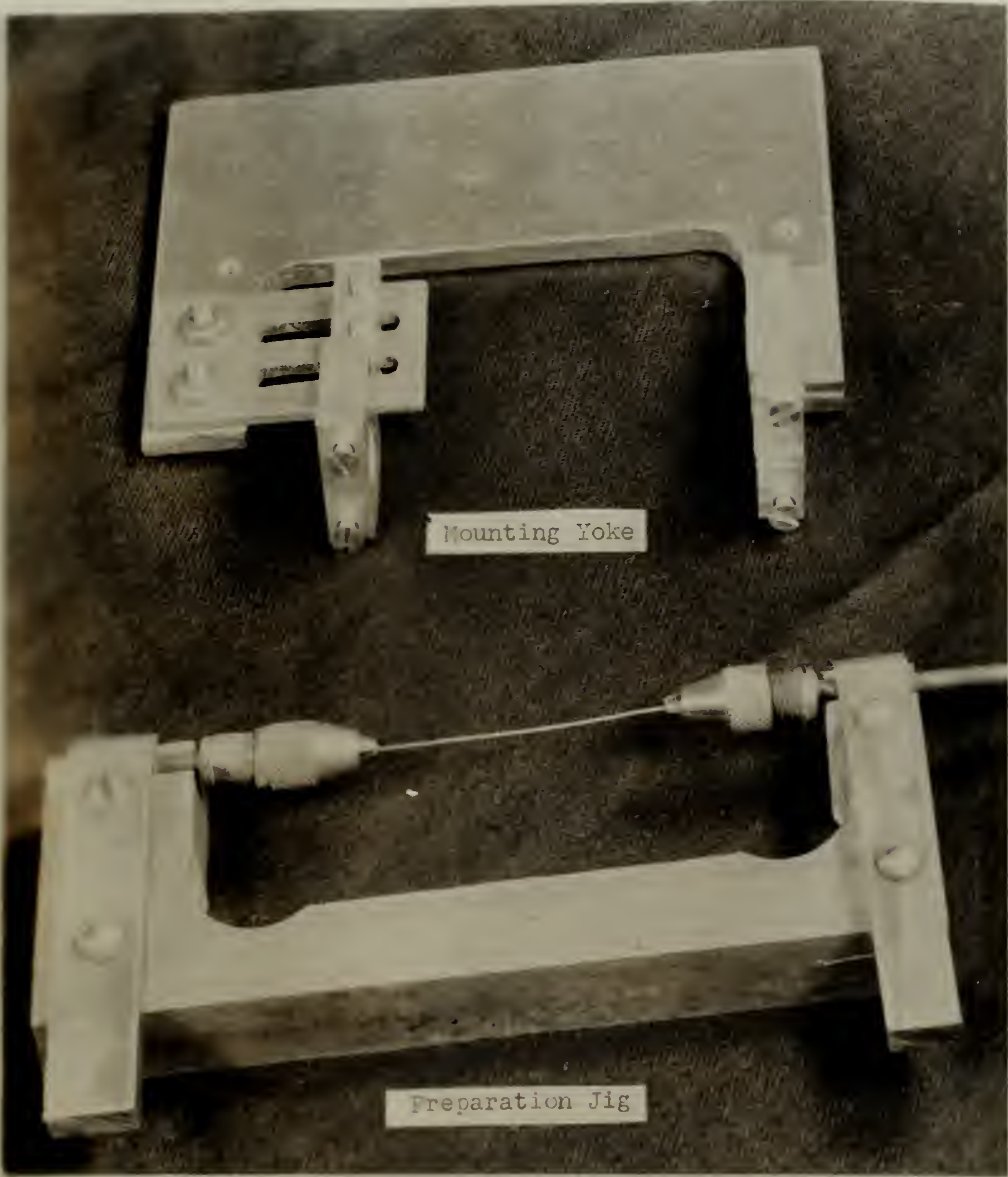
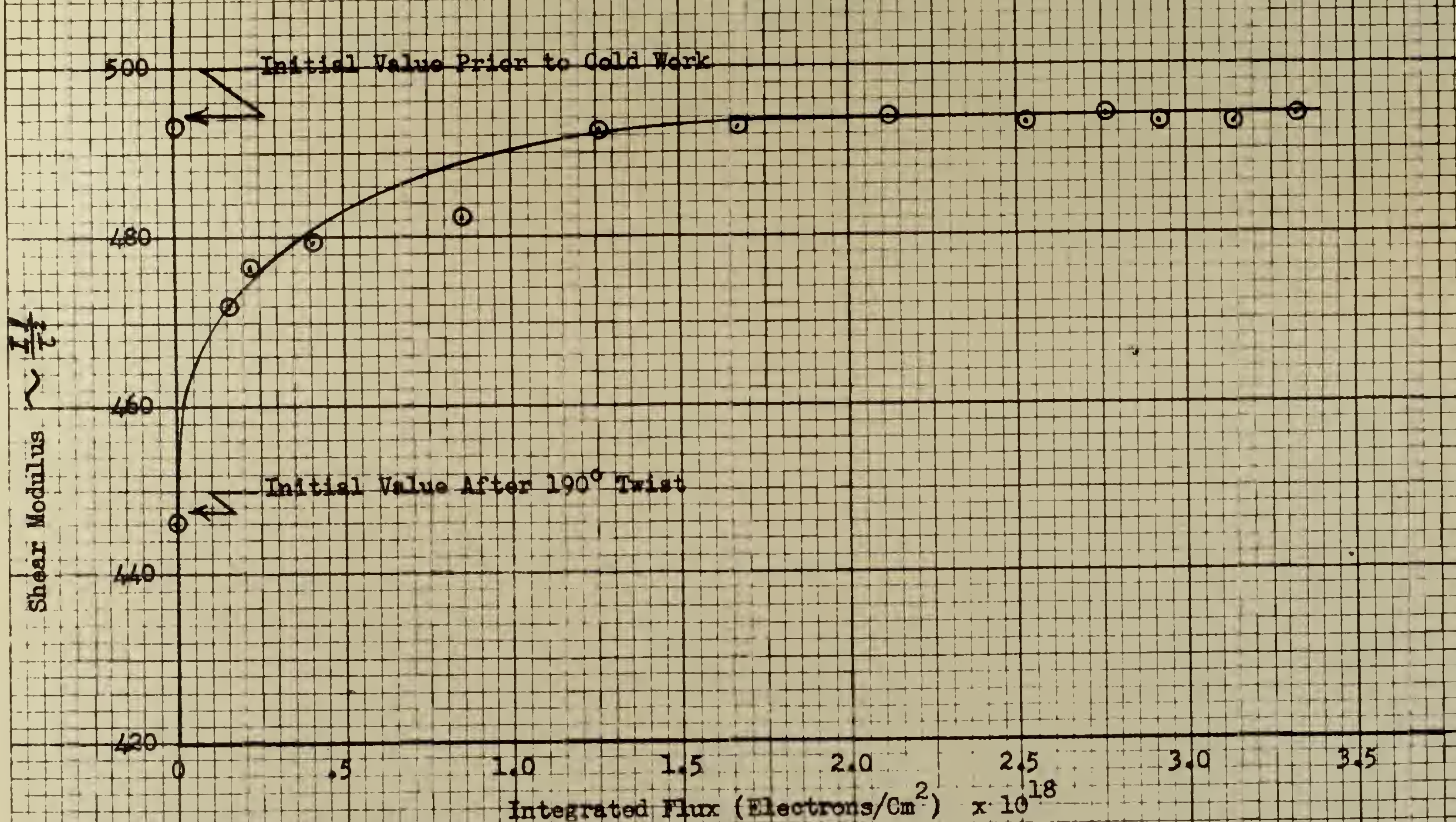



FIG. 7

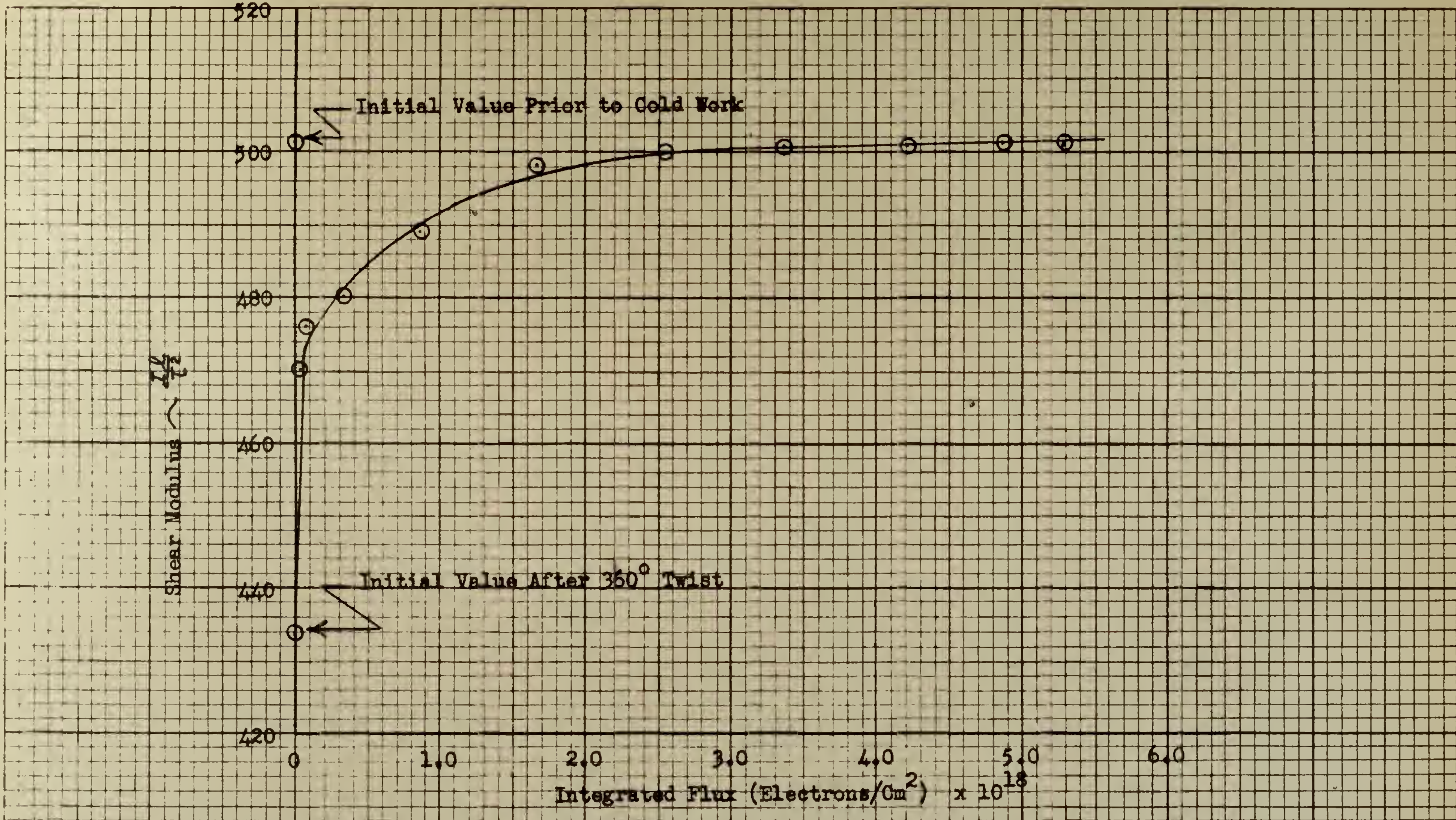
PREPARATION AND HANDLING EQUIPMENT

FOR SPECIMEN



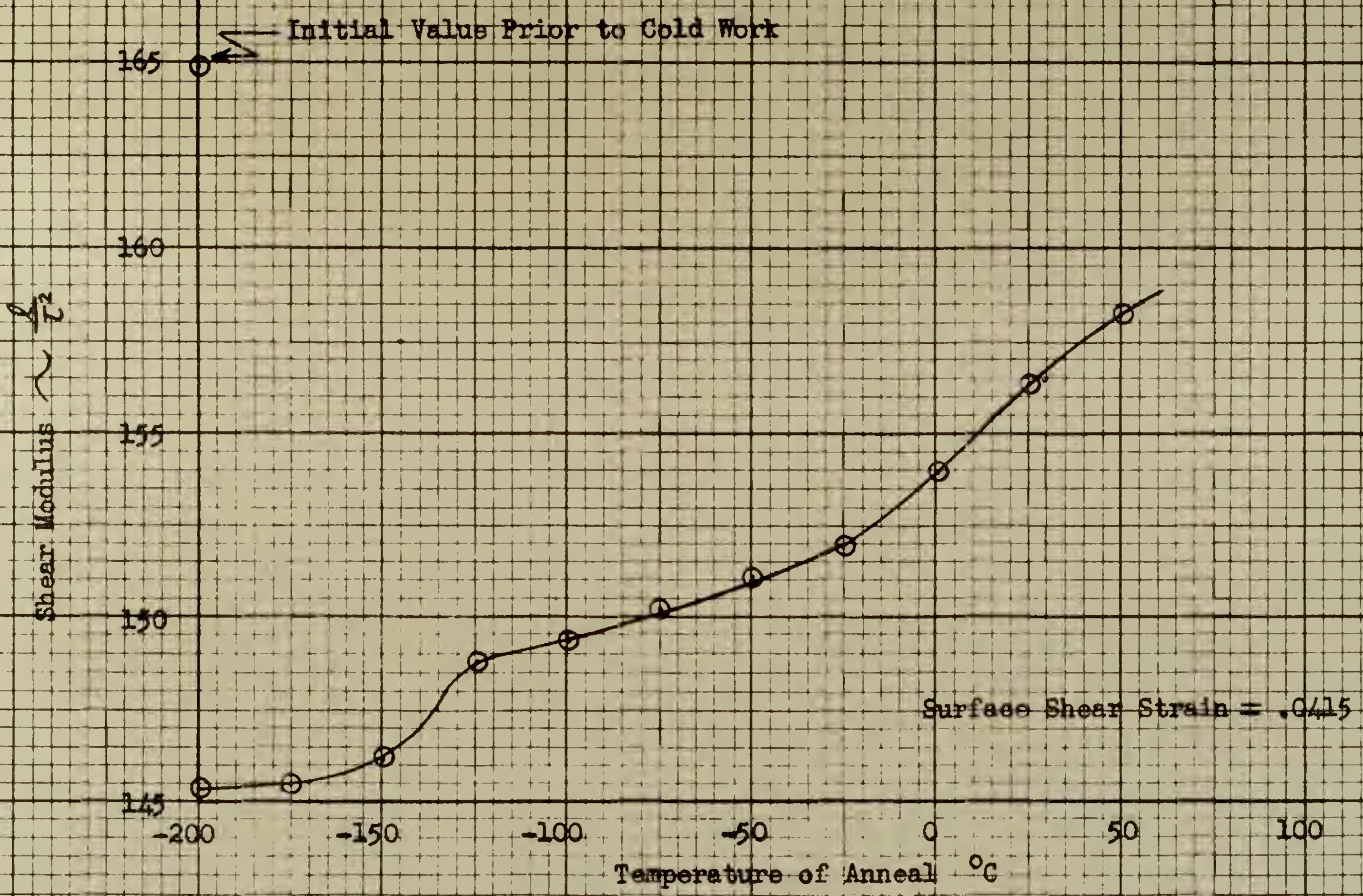

SHEAR MODULUS of COPPER AFTER COLD WORK
vs
INTEGRATED ELECTRON FLUX

FIG. 8



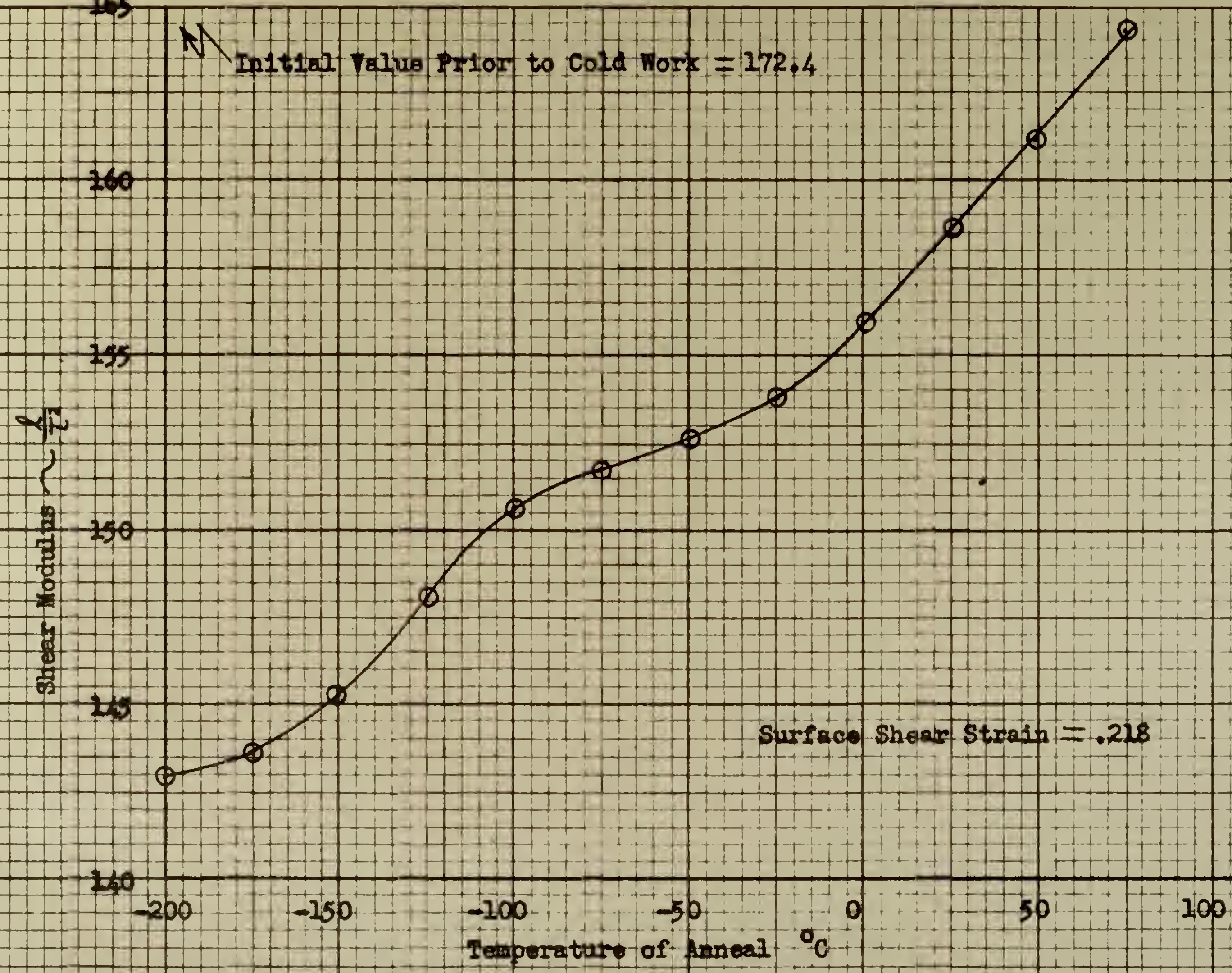

SHEAR MODULUS of COPPER AFTER COLD WORK
vs
INTEGRATED ELECTRON FLUX

FIG. 9



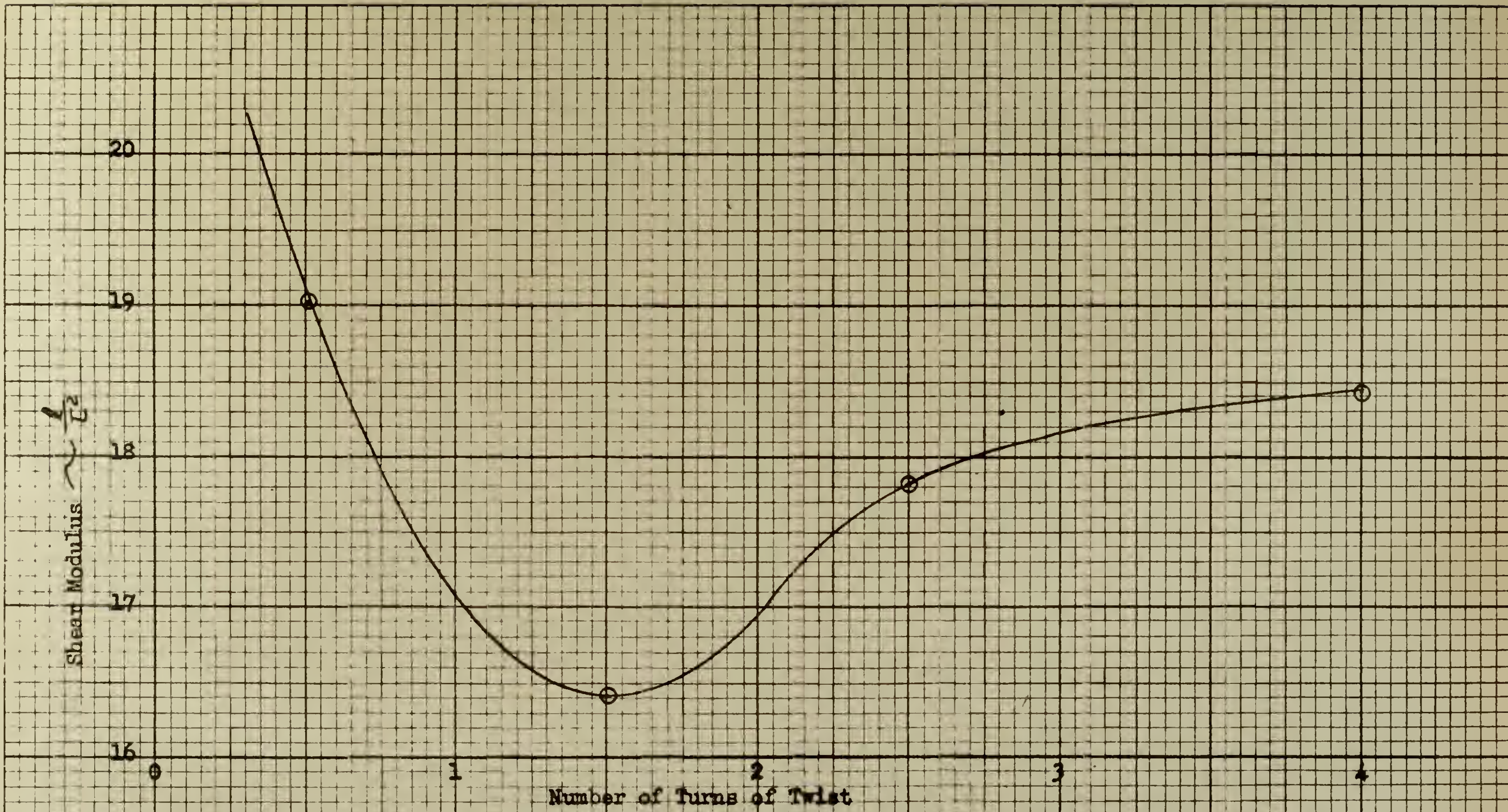

SHEAR MODULUS of COPPER AFTER COLD WORK
vs
ANNEALING TEMPERATURE

FIG. 10



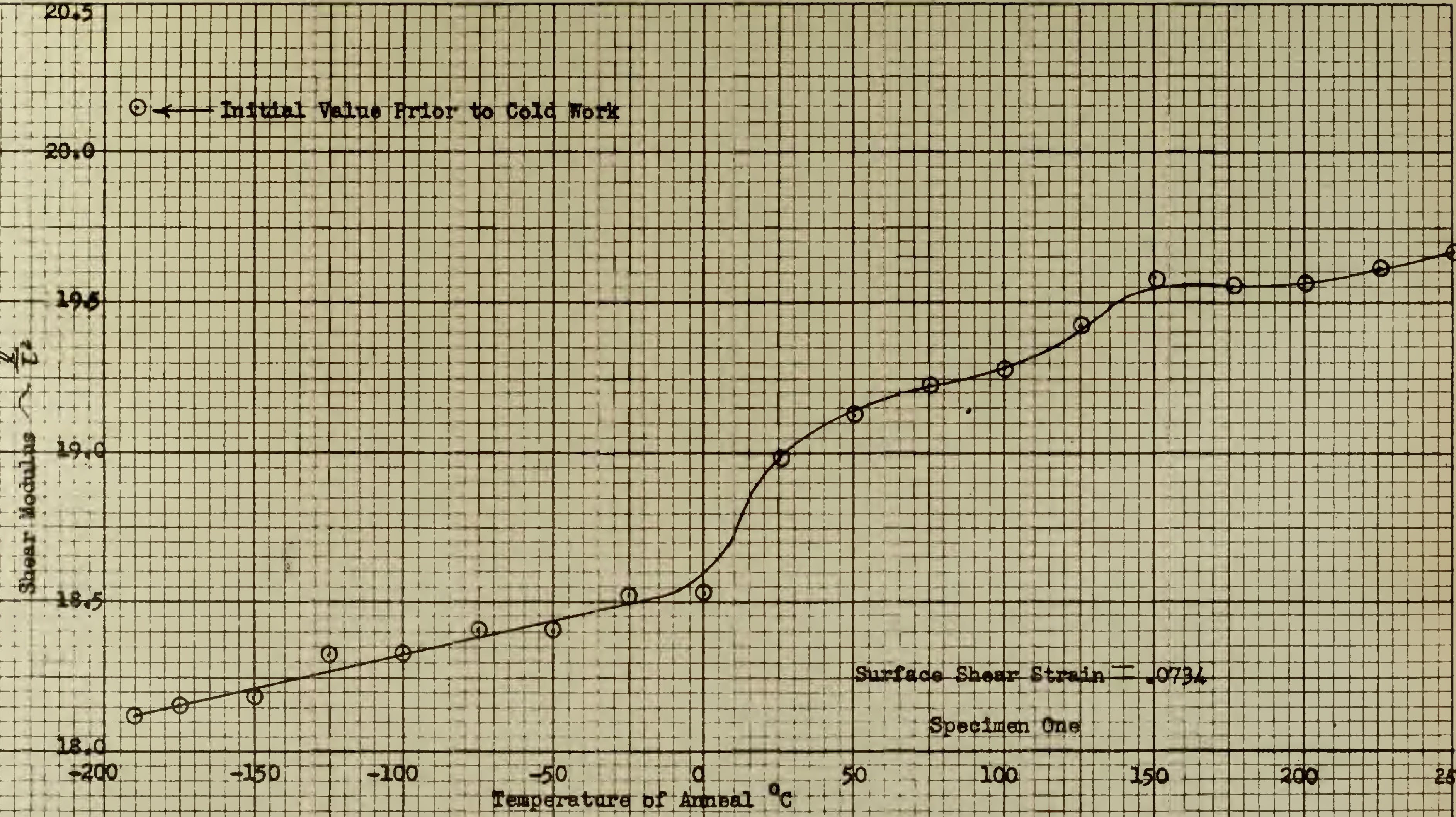

SHEAR MODULUS OF Cu AFTER COLD WORK
vs
ANNEALING TEMPERATURE

FIG. 11



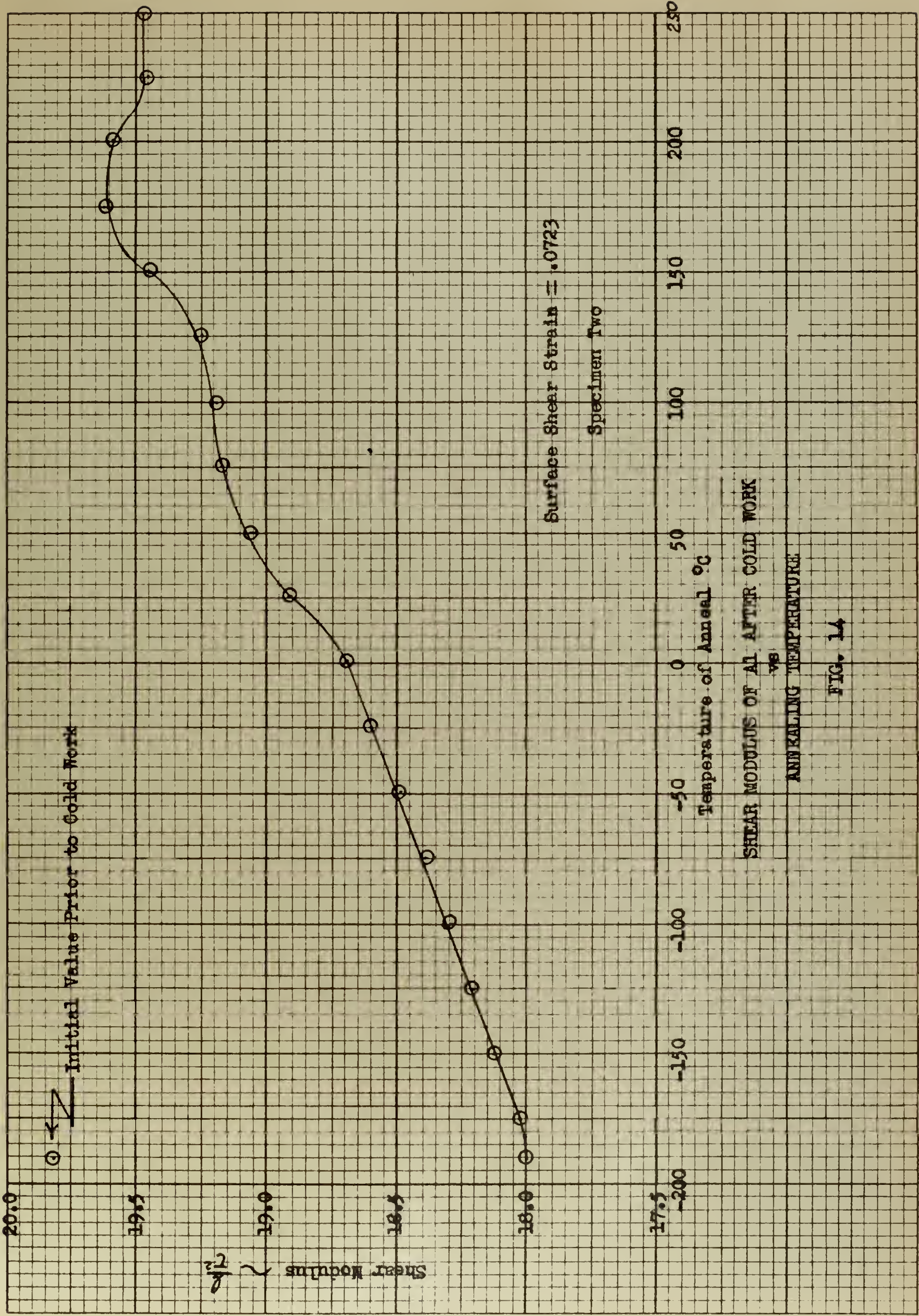

SHEAR MODULUS OF Al at -268.8 °C
vs
NUMBER OF TURNS OF TWIST

FIG. 12



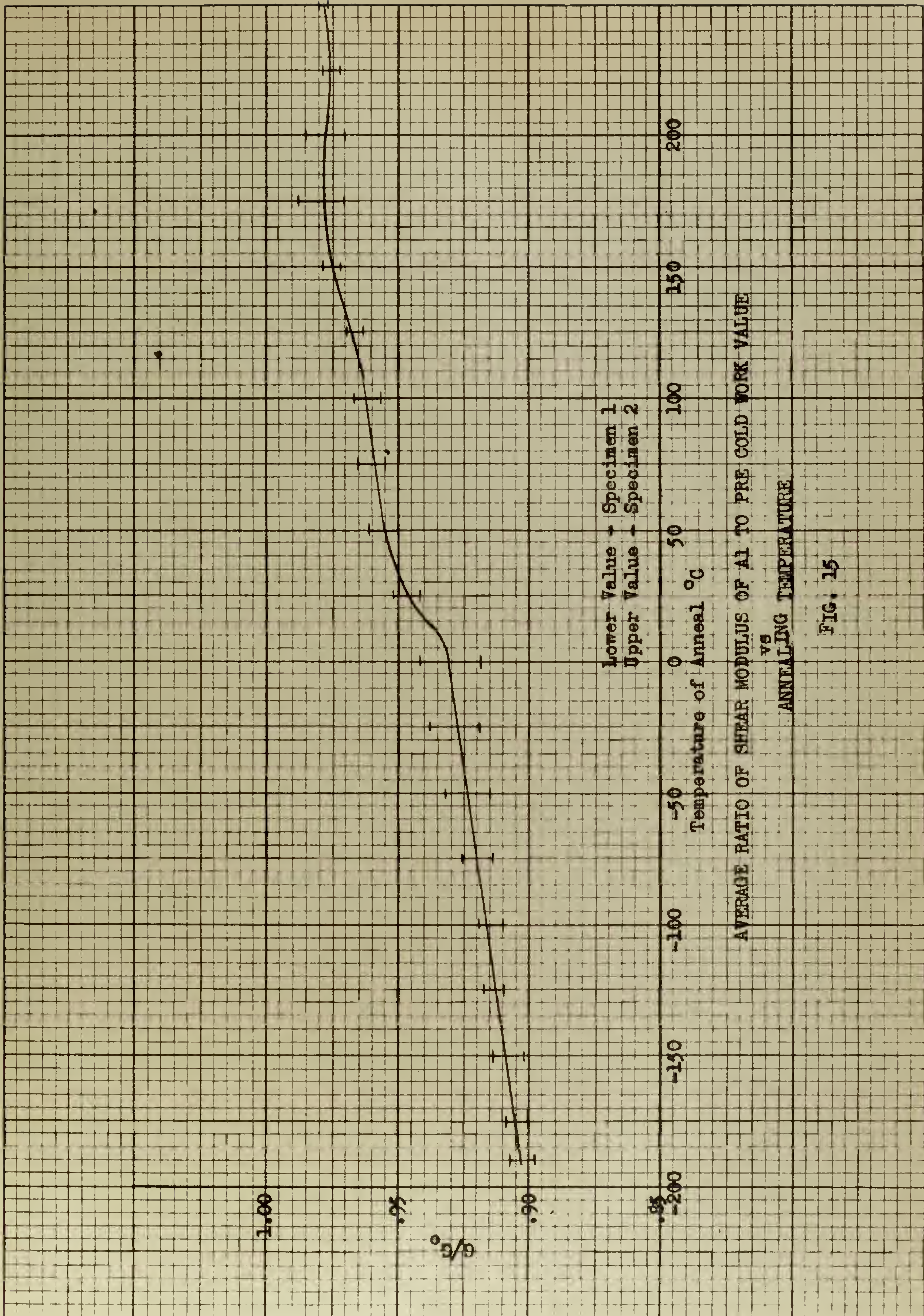

SHEAR MODULUS OF Al AFTER COLD WORK
vs
ANNEALING TEMPERATURE

FIG. 13




SHEAR MODULUS OF Al AFTER COLD WORK
vs
ANNEALING TEMPERATURE

FIG. 14




AVERAGE RATIO OF SHEAR MODULUS OF Al TO PRE COLD WORK VALUE vs ANNEALING TEMPERATURE

FIG. 15